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NOTE ON AN IMPROVED CHAIN-PACKED DISTILLING COLUMN [1]

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ABSTRACT

A description of an automatically controlled, chain-packed column capable of giving approximately 100 percent pure benzene and ethylene chloride, which boil 3.42° C. apart, from an initial charge of a 50 mole percent mixture of the two. This still is small enough to be installed in an ordinary sized laboratory.

In a previous paper [3] a detailed description has been given of the construction, operation, and efficiency of various forms of laboratory stills for the fractional distillation of liquids. A still with a 35-foot column packed with jack-chain had a higher efficiency than any of the other stills. Such a still is, however, expensive to construct and because of its height cannot be erected in an ordinary laboratory.

The purpose of this note is to describe a still with a chain-packed column of high efficiency, which can be erected in any laboratory of the usual height. The still is in every respect similar to those described in the previous paper except that a column packed with jewelers brass locket chain, size nos. 13 to 18, is employed.

A 3-liter still pot, supporting a column 2.5 cm in diameter and 250 cm long, requires approximately 3,600 feet of chain for filling. This filling should be uniformly distributed throughout, since any congested section in the column will cause flooding to occur before the most efficient operating conditions are reached. The over-all height of a still using this length of column is 322 cm, and the still can be readily installed in a room 344 cm high.

This still, running at a rate of 0.5 ml of distillate per minute, with a reflux ratio of 22 : 1, a vapor velocity of 7.57 m per minute, and having a "hold up" of 180 ml, readily separates a mixture of benzene and ethylene chloride. Starting with 1,500 ml of a 50 mole percent mixture of benzene and ethylene chloride, substances having boiling points differing by only 3.42° C., it was possible to obtain 450 ml of almost pure benzene as distillate and 350 ml of almost pure ethylene chloride as residue (fig. 1). Thus 53 percent of the charge was obtained as pure benzene and pure ethylene chloride. This percentage could be increased readily by either decreasing the amount of liquid held in the column or increasing the volume of the charge. In increasing the volume of the charge it is necessary to keep the evaporat-

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[3] Johannes H. Bruun and Sylvester T. Schicktanz, Laboratory Rectifying Stills of Glass, B.S.Jour. Research, vol. 7, p. 852, 1931.

ing surface of the liquid small enough so that upon brisk boiling the evaporating liquid does not cause continual flooding in the column. The 2.5-cm column was found to be approximately the right size to take care of the evaporation from the surface of the liquid exposed in a 3-liter round-bottom flask. Larger charges than this could be distilled satisfactorily through this column, if an elongated pot was used, thus keeping the surface area approximately the same.

In a column of this type the difference in pressure between the still head and the still pot is one of the controlling factors in obtaining the maximum efficiency. It was found that with a column 2.5 cm in diameter and 250 cm long the maximum efficiency was obtained when using a pressure difference of 26 mm of Hg, whereas in a smaller column, 2 cm in diameter, and of the same length, a pressure difference of 15 mm gave the maximum efficiency. In order to maintain this

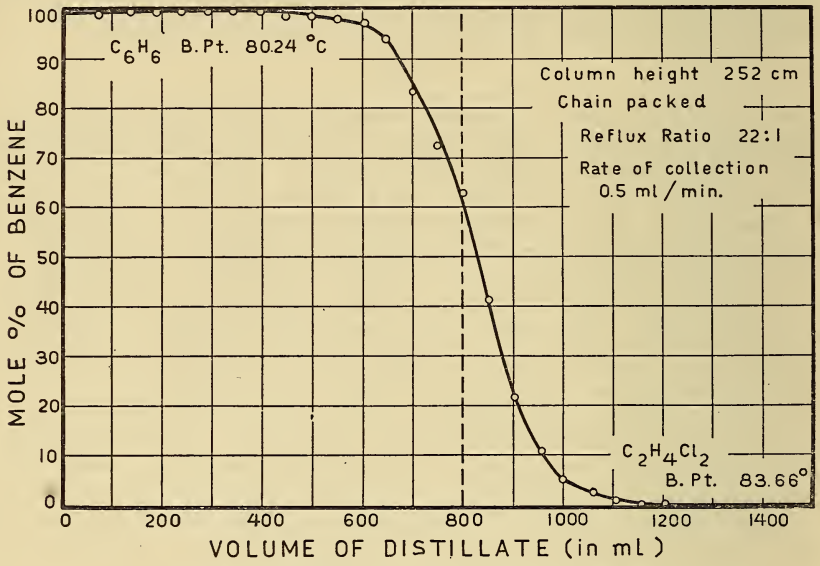

FIGURE 1.—*Efficiency curve for locket-chain still.*

Ordinate.—Mole percent of benzene in distillate.
Abscissa.—Total volume of distillate collected.

pressure at the value found most efficient for the still in question, the current in the coil, which heats the still-pot, is automatically controlled. The pressure difference is indicated by means of a differential manometer, one end of which is attached to the pot and the other to the still head. Two contact points are placed 1 mm apart in the high side of the manometer and one contact in the mercury proper. The two contacts control the high and low voltage regulators which are actuated by means of mercury relays. The voltage controls are nothing more than fixed resistances placed in the line, as illustrated in figure 2. When the mercury in the manometer (M) makes contact with the high contact point, a resistance (R_1) is thrown into series with the still-pot heater coil (S) by breaking the contact in relay (A), thereby decreasing the potential drop across the coil itself; and when the mercury breaks contact with the low point, the circuit through (A_1) is closed and a resistance (R_2) is thrown in parallel with rheostat (R_3)

which controls the heating coil, thereby increasing the potential across the heater.

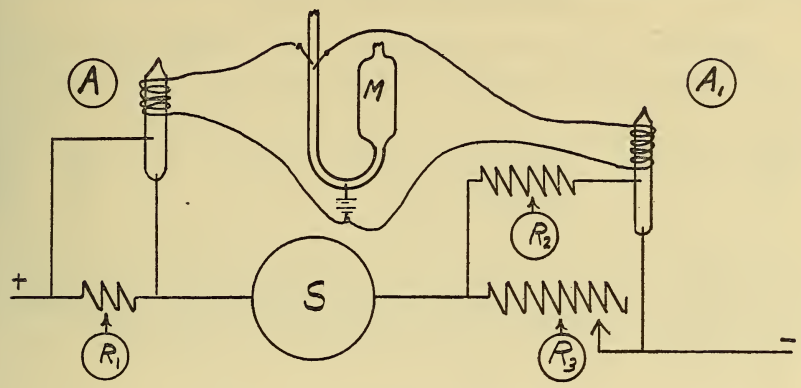

FIGURE 2.—*Diagram of automatically controlled heater unit for still pot.*

Mercury relays, without moving parts, were used since currents of from 4 to 6 amperes had to be broken. The relay is illustrated in figure 3; *A* is a 10-mm pyrex tube, *B* is the solenoid consisting of 600 ohms of enameled silk-covered copper wire No. 33, *C* and *C'* are two sealed-in tungsten contact points, and *D* is a soft-iron plunger. The relay is assembled and then evacuated and sealed off in order to prevent the mercury from fouling the contact points when operating under heavy loads.

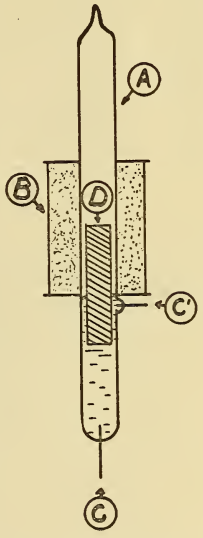

FIGURE 3.—*Diagram of mercury relay.*

To prevent sparking at the contact points, caused by the slow moving mercury boundary in the manometer, it was found desirable to use alternating current instead of direct current, thus obviating the expense of installing a vacuum tube hook-up. The reflux ratio is another important factor in operating a still efficiently. The greater the ratio the more efficient the separation. In the 2.5 cm column a 22:1 ratio gave initially 99.1 percent pure benzene ($n_D^{25.2}$ 1.4972 as compared to $n_D^{25.2}$ 1.4976 benzene initially used for the charge). An 11:1 ratio gave only 96 percent benzene.

The column should be run with no lateral heat loss, since the most efficient separation occurs when there is a uniform temperature gradient along the column. Hot and cold spots in the column cause flooding which decreases the efficiency. The same method of controling the temperature gradient is used on the packed column as was used on the plate stills.[4]

Table 1 shows the results obtained when using different types of columns and different packings.

[4] See footnote 3, p. 89.

TABLE 1.—*Separation of benzene and ethylene chloride using different distilling columns*

Type of still	Size of chain	Rate of distillation	Reflux ratio	Purity of initial fraction
		ml/min.		*Percent*
30-plate		1.0	10:1	78.0
Do		.75	20:1	84.0
Packed	22 jack-chain	1.0	10:1	80.0
Do	24 double jack-chain	1.0	10:1	83.3
Do	Locket chain	1.0	11:1	96.0
Do	do	.5	22:1	99.1

The efficiency is increased as we go from the jack-chain to the double jack-chain and to jewelers brass locket chain in accordance with the increase of effective surface exposed in the column.

WASHINGTON, April 13, 1933.